

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 4019

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OF SINGLE CRYSTALS OF AZ61X MAGNESIUM ALLOY

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SUMMARY

Exploratory tests were made of the stress-corrosion cracking of single crystals of solution-treated AZ61X magnesium alloy in distilled water, in 0.5 and 1 percent hydrofluoric acid, and in a salt-chromate solution consisting of 35 grams of sodium chloride and 20 grams of potassium chromate per liter. In most of the successful tests the basal plane was at an intermediate angle to the specimen axis; in all the corroders, these specimens failed at nominal tensile stresses in the range of 3,500 to 4,300 pounds per square inch; pronounced basal slip (plus accompanying modes of deformation) preceded cracking and failure started at an angle of 70° to 90° from the basal plane. Although the deformation processes were not studied in detail, the path chosen by the crack was attributed to mechanical rather than electrochemical factors and the possible importance of the prior deformation, which has not been considered previously, was pointed out. The presence of a stress producing macroscopic slip was found a sufficient condition for cracking in the presence of a suitable corrodent; whether it is also a necessary condition was not established. The present work did not provide any data with which either the film-rupture theory of stress-corrosion cracking (electrochemical nucleation and propagation) or the mechanical theory (electrochemical nucleation and mechanical propagation) could be critically evaluated. Basal failure occurred in only one of three cases where the basal plane was at a relatively high angle to the specimen axis and therefore disposed favorably for this type of failure; examination of these data led to a tentative explanation for the reported tendency for basal failure in polycrystals.

INTRODUCTION

In those magnesium alloys susceptible to stress-corrosion cracking, the failure is transgranular under many conditions (ref. 1). The present work was undertaken to investigate the factors influencing the path taken by a transgranular crack and consisted of a preliminary study of the failure of stressed single crystals of various orientations of the

AZ61X alloy exposed to several corrodents. Although this study had to be terminated abruptly and the results were therefore not so comprehensive as originally planned, some important conclusions could be drawn.

Stress-corrosion experiments which will yield fundamental information are difficult to design since no firm opinion can now be given as to the mechanism by which a stress-corrosion crack is nucleated or propagated. Intergranular failure appears to be the simpler case since the course of the crack follows an anodic path preexisting in the alloy structure (ref. 2). In the case of transgranular failure, there is, in general, no unique preexisting anodic path (except for those running along subgrain boundaries and these have not been shown to be important). Transgranular failure can be interpreted in terms of the essential ideas of the generalized theory proposed by Mears, Brown, and Dix (ref. 2) in the following way. Electrochemical corrosion produces local penetrations, the rate of advance of which soon drops to a low value as films form on the surfaces of the crack or pit; the stress concentration at the tip of the crack produces local plastic deformation which results in rupturing of the film at that point and the continual exposure of film-free anodic metal thus permits electrochemical attack to continue. This mechanism leads to the conclusion that, in a given grain, a transgranular crack will propagate in a direction determined by the location of the regions over which the film is ruptured and thus by the geometry of plastic deformation at the crack tip. A quite different possibility is that the crack propagates mechanically as suggested by Keating (ref. 3) and Harwood (ref. 4). This mechanism postulates that the crack can be propagated between structural barriers in the metal by stress alone, the function of corrosion being the formation of the original crack as well as the reactivation of the crack after its progress has been stopped by a barrier. It is important to note that, although electrochemical action is an indispensable feature of both mechanisms, the direction in which a transgranular crack will propagate would in either case depend on the mechanical properties of the metal at the crack tip and may or may not vary with crystallographic orientation depending on how strongly anisotropic these properties are. Attention is thus directed to mechanical factors. It is difficult to make precise observations of the mechanical behavior at the crack tip; however, the study of single crystals may prove fruitful since the variation in stress-corrosion behavior can be observed as a function of crystal orientation, hence, of varying mechanical properties.

Studies of magnesium-alloy single crystals were first made by Graf (ref. 5) on solution-treated specimens of AZ61 alloy under a stress of about 4 kilograms per square millimeter (6,000 psi), which was slightly in the plastic range, in distilled water. Graf noted that the cracks always ran more or less parallel to the plane of maximum tensile stress but few conclusions can be drawn since he studied only crystals where the basal plane was approximately parallel to the stress axis. It may

be noted, however, that he made a careful study of polycrystalline specimens which showed that the stress-corrosion susceptibility was not significant unless the stress was sufficient to produce at least a very small plastic deformation (somewhat less than 10 percent of the elastic strain). A similar conclusion may be drawn from unpublished work done by the Dow Chemical Co. which showed, for a variety of polycrystalline magnesium alloys tested in the standard salt-chromate solution (35 grams of sodium chloride plus 20 grams of potassium chromate per liter), that the minimum stress required to produce significant stress-corrosion susceptibility was practically equal to the minimum stress at which the solution potential of the alloy showed a (transient) anodic shift, the anodic shift indicating that enough film-free surface had been exposed by plastic deformation to give a detectable effect.

While the present work was in progress, other observations of the path of failure were made by Priest, Beck, and Fontana (ref. 6) in coarse-grained specimens of the (controlled purity) AZ61X alloy in the standard salt-chromate solution under high stresses (substantially above the yield stress, which was 32,000 psi). They found that the path of failure tended to follow the basal plane and postulated that the preferred path direction was due to electrochemical factors, that is, to the directionality of the FeAl platelets which, as the work of Heidenreich, Gerould, and McNulty (ref. 7) strongly suggests, serve as cathodes in transgranular failure. The FeAl fine structure was assumed to be lying parallel to the basal plane.

The results of the present work show that failures often start and run on planes other than the basal plane and are thus inconsistent with the hypothesis of Priest, Beck, and Fontana. The present results are consistent with the view that deformation or fracture mechanisms play a primary role in determining the course of a transgranular crack.

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EXPERIMENTAL PROCEDURE

The material studied was 1/16-inch AZ61X magnesium alloy sheet supplied in the annealed condition by the Dow Chemical Co. who furnished the following analysis: aluminum, 5.6 percent; zinc, 0.76 percent; manganese, 0.33 percent; calcium, less than 0.01 percent; copper, 0.004 percent; iron, less than 0.001 percent; nickel, 0.0006 percent; lead,

0.012 percent; silicon, less than 0.01 percent; and tin, less than 0.001 percent. Strips of this alloy were stacked and milled to the form of tensile specimens about 4 inches long with a reduced section $1\frac{1}{4}$ inch wide and $1\frac{1}{4}$ inches long and a bolt hole at each end.

Considerable effort was required to develop a reliable procedure for preparing and handling single-crystal specimens. The heat-treating technique developed involved sealing the specimen in a Pyrex tube after admitting air at a pressure of about $1\frac{1}{4}$ atmosphere. When the specimen was heated, the oxide and/or nitride film formed served to minimize the rapid evaporation of magnesium and deterioration of the specimen which would otherwise have occurred. To prepare single crystals, some of the blanks were subjected to a plastic strain in tension of about 0.25 percent and the machined surfaces were polished and etched to remove the cold-worked layer. The specimen capsules were annealed by raising the furnace temperature 50°C per day from 300° to 480°C or else they were passed through a traveling furnace set at 480°C at the rate of 1 inch per day. On the average, the latter procedure gave better results. Although a large number of the crystals used had been strained before annealing, strain was not found necessary for the growth of large grains which presumably resulted from the exaggerated growth of a few grains in the specimens at high temperatures rather than from a recrystallization process. About half the annealed blanks had grains in the test area coarse enough to be useful, but a number of these were unsatisfactory because of small included grains in the central portion of the reduced section or because the specimen was not straight.

Several more-or-less typical specimens are shown in figure 1. In a few only one grain covered the test length (center specimen). In many, a short grain could be masked out to achieve the same result (bottom specimen). A number of specimens with two or three grains in the reduced length were tested to determine through which grain failure would occur. These specimens had grains of roughly equal length; the multigrain specimen shown at the top in figure 1 is unrepresentative in this respect.

Annealing and straining-and-annealing methods did not yield coarse-grained specimens in the lots furnished of AZ31X and M-1 sheet originally intended for inclusion in this study.

The orientations of the AZ61X crystals, determined from back-reflection Laue photographs, were scattered over the whole orientation range. Many of the AZ61X crystals were tested as air-cooled from the crystal preparation anneal. A number were subsequently annealed at 350°C for 24 hours and water-quenched. In order to prevent distortion during this treatment, the crystals were clamped between graphite blocks before encapsulating.

The dead-weight loading apparatus for stress-corrosion testing is shown in figure 2. The specimen A is held between steel clamps (later AZ61X clamps) and the load is transmitted by steel strips C looped over self-aligning ball bearings B, an arrangement which enabled fair initial axiality of loading to be achieved (the stresses on opposite edges of the specimen for loads in the elastic range were at worst within 15 percent of each other as determined in several runs on a polycrystalline dummy with the aid of Huggenberger extensometers). Axiality would have been important if failures had occurred under stresses within the elastic range, but no failures were observed until the specimen was well within the plastic range. Axiality cannot then be maintained because of the gross inhomogeneity of the deformation of hexagonal single crystals, especially under the present conditions, where (1) the ratio of test length to specimen width is low so that bend gliding at the ends of the test length can influence a substantial portion of the test length and (2) a grain outside the test length may be so much more favorably oriented for slip that it is the first to slip, thus making axial loading of the grain under test impossible. For these reasons, many of the failures observed may have been initiated by local stresses much higher than the nominal stress.

The corrodent G (fig. 2) was held in an acrylic cup F fitted with a neoprene plug E which slid over the beeswax-rosin masking D at the ends of the specimen, this joint and the steel clamps being covered with petrolatum for added protection. Wax masking was also applied at the upper end of the specimen so that the specimen was not exposed at the air interface. The cup was covered during the test to minimize evaporation but to permit access of oxygen. At the start of each test, the corrodent was always added before applying the load. The specimen had previously been electropolished in the polishing bath of hydrochloric acid and Cellosolve in order to facilitate the observation of cracks and strain markings during the test.

The experimental difficulties encountered in these tests are described later. Pilot tests made to aid in selecting suitable heat treatment and corrodent are also described below.

The stress-corrosion experiments described herein were concerned only with the variables of stress, crystal orientation, and type of corrodent. Microstructural variables were not included; all specimens were essentially solution-treated. Consideration of the subgrain structure as an additional variable had been planned initially. It was found, however, that the crystals did not have pronounced substructures; the orientation spread indicated by the size of the Laue spots was very small. A brief attempt was made to reveal subboundaries by etching. Of the first five microetchants listed in the Metals Handbook (ref. 8), the glycol-acetic-acid-nitric-acid solution etched low-angle grain boundaries (orientation difference estimated as several degrees) as did the

picral—acetic-acid etchant described by George (ref. 9), but neither revealed any distinct subgrain-boundary structure. At this point, work in this direction was halted since the stress-corrosion test results showed that failure occurred after pronounced plastic deformation and it seemed likely that this produced structural defects substantially more severe than those provided by the original subgrain structure which thus did not appear to be a primary factor in this work.

EXPERIMENTAL RESULTS

Pilot Tests on Polycrystalline Specimens

As a guide in selecting test conditions, a number of constant-deflection bend tests were made using $1/4$ -inch-wide $6\frac{1}{16}$ -inch-long polycrystalline strips held in 5.61-inch-long phenolic jigs which produced initial stresses above the yield strength in the central region of the strip. The specimens were pickled before testing for 20 seconds in 10-percent nitric-acid solution. The results of the investigation of the solution-treating temperature yielding greatest susceptibility to stress corrosion using the standard salt-chromate solution (35 grams of sodium chloride plus 20 grams of potassium chromate per liter) are given in table I. A wide scatter is inherent in this type of test since whenever the time to failure is substantial, stress relaxation in the specimen tends to make the failure time even longer. The data in table I are therefore not considered to show any significant variation of susceptibility with annealing temperature in the range studied. A similar result was obtained by Perryman (ref. 1) for an experimental 5-percent-aluminum alloy.

It was of interest to survey single-crystal behavior in several corrodents producing relatively rapid failures without heavy general corrosion. Fluoride solutions were suggested by some of Perryman's results and some of these are compared with strip test results obtained using salt-chromate solution and distilled water which are given in table II. In the salt-chromate solution, a crack is observed after a very short time and failure occurs soon thereafter. In distilled water, a number of cracks formed after a few days and here also the specimens failed fairly soon after the appearance of cracks. However, in the fluoride solutions, fine, perhaps shallow, cracks formed not long after immersion, and in many cases these progressed very slowly so that failures did not always occur in reasonable times even when the stress in the specimen had not relaxed too far. The susceptibility in the 0.5 percent hydrofluoric acid seemed sufficient, however, and this solution was used for some of the single-crystal tests since the film it formed over the specimen surface was clear and permitted better observation of the strain

markings developing during the test than did the films formed in distilled water or salt-chromate (also used) which were dark and patchy. In all three corrodents, the failure of the polycrystalline strips was transgranular.

Single-Crystal Tests

General remarks.- Stress-corrosion tests using the arrangement shown in figure 2 were first made on a few preliminary coarse-grained specimens by increasing the stress stepwise until failure occurred. These showed plastic deformation to be important, aside from its effect on the axiality of loading, in that it led to experimental difficulties with crystals of some orientations and in that failure did not occur until it was observed. Although the modes of deformation of this alloy were not studied in detail, it was verified that the major mode of deformation in favorably oriented crystals was basal slip and many of the observations can be described in terms of this type of deformation. The observations of this investigation were consistent with the critical resolved-shear-stress law of Schmid and Boas (ref. 10) which states that (basal) glide begins when the resolved shear stress reaches a critical value τ_c . The initial shear stress is calculated from the relation $\tau = \sigma \sin \chi_0 \cos \lambda_0$ where σ is the tensile stress, χ_0 is the initial angle between the basal plane and the specimen axis, and λ_0 is the initial angle between the specimen axis and the nearest of the type I diagonal axes (which are assumed to be the possible slip directions). For the present purposes, the error is small if it is assumed that $\lambda_0 = \chi_0$ and the tensile stress needed to produce basal slip can then be written

$$\sigma_c = 2\tau_c / \sin 2\chi_0 \quad (1)$$

The critical tensile stress σ_c is thus a minimum at 45° and increases symmetrically on either side of this angle so that at 15° or 75° it is twice the value at 45° . If plastic deformation is necessary for failure, then a high tensile stress is required when the grain under test has a high or a low orientation and this stress may be sufficient to produce deformation in grains at the ends of the test length underneath the wax (such a situation might arise in the bottom specimen of fig. 1); the deformation of these grains could allow the corrodent to leak under the wax and either (1) reach the steel clamp resulting in rapid local general corrosion and mechanical failure near the clamp when the specimen cross section had been sufficiently reduced or (2) achieve the same result because of local general corrosion in the oxygen-depleted area or at the solution-vapor interface underneath the wax. Two failures (in the salt-chromate solution) attributable to the first situation and four (three

of these in 0.5 percent hydrofluoric acid), to the second were obtained, and all of these were cases where the orientation was in the low (25° or less) or high (greater than 65°) range. For these reasons, only a few specimens of the extreme orientations were successfully tested, and thus most of the data refer to the situation where basal slip was the dominant mode of deformation. It should be noted that deformation markings which appeared to be twins were observed but their character and crystallography were not verified; it is also possible that nonbasal slip occurred. These could have accounted for large local strains even though every case where substantial elongation (say 5 to 10 percent) of a grain was observed was associated with basal slip. In the present work, the first indications of basal slip (weak slipbands) were noted at resolved shear stresses in the neighborhood of 1,500 psi which is in good agreement with the average critical shear stress of 1,640 psi (1,153 g/sq mm) observed for AZ61 crystals by Schmid and Siebel (ref. 11).

Significant information was provided by one specimen which failed because of the electrochemical coupling at the steel clamp. This had been air cooled from 480°C and had an orientation angle of 19° ; it was stressed in salt-chromate solution without failure for $4\frac{1}{2}$ weeks at

2,300 psi, then for 2 weeks at 2,850 psi, and then for 1 week at 3,260 psi (all without evidence of plastic deformation) before failing outside the test area at 3,850 psi. While this did not provide a critical test of whether failure can occur at all in the elastic range, it did indicate that the stress-corrosion life of these specimens was at least substantial at stresses which did not produce plastic flow.

For subsequent tests, the specimens were stressed stepwise to failure at intervals of 1 week by starting at a load too low to give visible deformation or strain markings (usually 3,000 psi, in a few cases 2,000 psi) and then entering and moving up in the plastic range in steps of about 500 psi. Since failures were not observed below the plastic range, this procedure provided assurance that the failure stress was always approached from the low side and enabled the nominal tensile-stress values obtained to be used as a measure of comparative susceptibility. In a few cases (these are indicated in tables III(a) and III(b)), the load was increased in shorter time or larger stress intervals, but here also the specimen was under observation while the load was being increased in order to be sure that relatively small stress increments would be used in moving into the plastic range.

Importance of plastic deformation.— One type of observation indicating the importance of plastic deformation is shown in figure 3. Some time after loading the specimen shown, a band where basal slip (orientation of basal plane, 31°) was particularly heavy had developed and it was in this band that a crack developed and failure occurred.

Analogous behavior was observed in the two- or three-grain specimens described in table III(a). No tendency for failure to be intergranular was noted. In specimens 7 and 23, failure occurs in the grain most favorably oriented for basal slip (the one having the orientation closest to 45°) and evidence of considerable basal slip was observed in the failure grain prior to the appearance of a visible crack. Although in specimen 6 failure also occurred in the more favorably oriented 8° grain, this specimen represents a different situation since the resolved shear stress was too low for basal slip; strain markings could not be studied because of the dark patchy film resulting from the distilled water exposure, and there was little if any change in shape of the failure grain. However, X-ray photographs of areas removed from the region of failure indicated the failure (8°) grain to have suffered substantial lattice distortion while the other (1°) grain had not. X-ray photographs of specimen 7 (fig. 4) and specimen 23 showed the same conditions except that the distortion of the failure grain was greater. Thus, in these three specimens, in all of which the failure ran at a high angle to the basal plane, the observations indicate the stress-corrosion cracking to be strongly associated with the plastic properties of the alloy.

In specimen 12, failure along the basal plane occurred in the 75° grain after a few faint unidentified strain markings had been noted; additional markings (some appeared to be twins) and slight distortion (fig. 5) appeared in the course of failure. No X-ray study was made, and no statement can be made concerning the influence of plastic deformation in this case of basal failure.

It may be noted that one unfavorably oriented 82° specimen (water-quenched), which failed in the 0.5 percent hydrofluoric acid through the generally corroded area underneath the wax, withstood a stress of (at least) 5,500 psi. It is not believed that the cathodic protection afforded by the anodic areas under the wax substantially affected the body of the specimen.

Comparison of behavior in various corrodents.— The behavior of specimens having orientations in the 29° to 37° range, all of which suffered a substantial amount of basal slip, was quite similar in all of the corrodents employed (table III(b)). In each case, pronounced slip preceded crack formation. The variation in the nominal stress for failure cannot be considered significant in view of the nonaxiality of the loading. Specimen 33, which was stressed in accordance with the customary increment schedule of 1 week at 500 psi, shows that, in the absence of a corrodent, these crystals will withstand stresses substantially above the stress-corrosion limit for extended periods without any indication of cracking.

It may be noted in table III(b) that distilled water and hydrofluoric acid favored the formation of a number of additional cracks

while only the crack which led to failure formed in the salt-chromate solution test, similar to the behavior of polycrystalline specimens noted in table II. It was pointed out to the authors by the manufacturer that this behavior may be interpreted on the basis that the throw of protective current from the first crack formed in a highly conductive environment (salt-chromate solution) is great enough to inhibit crack formation elsewhere on the specimen, while in the poorly conductive environments (water and fluoride solutions), the throw is more or less restricted to the vicinity of the crack.

In the one successful single-crystal test in salt-chromate solution, failure occurred a half hour after the final load increment had been applied, but no statement can be made as to whether single crystals would always show the rapid failure in this solution characteristic of polycrystalline specimens stressed above the yield strength.

No direct comparison was made of the behavior of the air-cooled as compared with that of the water-quenched specimens; the data in tables III(a) and III(b) do not suggest any large difference.

Plane of failure.- Since the final stages of failure in these experiments were considered to represent simple mechanical failure under a high stress, only the first-formed part of the failure surface was considered to provide significant information on the path of a true stress-corrosion crack. The first part of the failure was often relatively smooth with irregular fine structure and sometimes very shallow but well-defined steps. It was more or less macroscopically plane, and this plane was related to the crystal orientation as expected in view of the observed influence of plastic deformation; in view of the deformation preceding failure, it was improbable that the crack surface would have been microscopically plane in any event.

In specimens exhibiting basal slip, the crack ran initially at a high angle, estimated to lie in the range 70° to 90° , to the basal plane (figs. 3 and 6) in all the corrodents used. This was also noted in the low-orientation specimen 6 (table III(a)). It was not determined whether the plane of failure in these specimens was a definite crystallographic plane; in many cases such a determination would have been subject to uncertainties arising from the distortion of the failure grain or to the fact that the failure was stepped or uneven.

Where pronounced basal slip was observed, a number of short edge cracks was often found (usually these were not observed until the main crack was well developed) and in a number of cases these were associated with the unidentified strain markings which ran across the basal slip traces (fig. 6). Many of the unidentified markings were found unaccompanied by cracks and it is thus presumed that these markings preceded the cracks rather than followed them.

The only failure to start parallel to the basal plane within the error of the orientation determinations occurred without pronounced basal slip in specimen 12 (table III(a)) in the 75° grain (fig. 5); it is possible that one factor favoring basal failure here is the high orientation angle which corresponds to a tensile stress across the basal plane almost equal to the maximum tensile stress. It is postulated that basal failure did not occur in specimen 23 (containing a 71° grain) and in specimen 7 (containing a 61° grain) because the alternative mode of failure involving prior basal slip could be produced at a lower applied stress. Thus, in specimen 23, the 29° grain deformed and failed first; in specimen 7, the 61° grain itself suffered basal slip and failed by the nonbasal mechanism; in specimen 12, the 5° grain was too unfavorably oriented to deform at the stresses involved, and the 75° grain had not yet reached the critical shear stress for basal slip so that, for this orientation, the alternative mechanism of failure was unable to precede the basal mechanism.

With regard to basal failure, it is perhaps noteworthy that the failure surface of specimen 32 in salt-chromate solution (table III(b)) showed a branch nearly parallel to the basal plane.

DISCUSSION

The film-rupture theory of stress corrosion requires that plastic deformation take place at least to the extent that the metal just under the crack tip continues to deform, and, because of the stress concentration under the crack, this deformation is presumed to be possible under stresses somewhat below the unnotched yield strength and thus in the absence of deformation detectable by the usual methods. Since the present work did not establish that failure does not occur at all below the macroscopic yield stress, it did not furnish a critical test of this theory. The present work indicates that a stress-producing macroscopic deformation of a single crystal of AZ61X magnesium alloy is a sufficient condition for transgranular stress-corrosion failure in the presence of a suitable corrodent. In the nonbasal failures, some deformation always preceded crack formation. Macroscopic inhomogeneity of this deformation would produce higher stresses in some parts of the specimen. However, microscopic inhomogeneities are likely to be more important; for example, it is likely that dislocation pileups will affect local concentration of the applied stress by at least several orders of magnitude under the conditions of these tests. It is also likely that lattice vacancies are produced during deformation. Thus, it is not unreasonable to suppose that a crack is nucleated much more easily in the deformed regions than in the unstrained material. Further, the deformation of these crystals involved substantial plastic bending, and the resultant strain-hardening could facilitate the mechanical propagation of a crack by inhibiting competing plastic processes. These considerations suggest that one of

the functions of plastic deformation not previously considered may be an alteration of the structure of this alloy which enhances or produces susceptibility to stress corrosion. This process can operate in connection with either of the two theories of the stress-corrosion mechanism considered; the mechanical factors involved in the present work are in a general way consistent with either theory.

As to the difference between the present data and the observation of Priest, Beck, and Fontana (ref. 6) that failure tended to be basal, the considerations discussed above in the section entitled "Plane of failure" may offer a clue. The present work shows that there is at least one mechanism of nonbasal failure and this involves basal slip (plus other accompanying deformation processes), which is produced at relatively low stresses in single crystals. It is then attractive to suppose that, in the tendency for basal failure observed by Priest, Beck, and Fontana in coarse-grained polycrystalline specimens, the essential circumstance was that the basal slip of each grain was severely limited by the constraints due to the presence of the surrounding grains so that the mechanism involving slip followed by nonbasal failure was unable to come into play. In this view, the predominant mechanism of failure in polycrystals may differ from the predominant mechanism in single crystals, except for single crystals in a specific orientation range.

While the present work does not provide information on the role of plastic deformation in basal failure, Priest, Beck, and Fontana showed that a wave of plastic deformation precedes an advancing transgranular crack in a polycrystalline aggregate where failure tends to occur along the basal plane. This, together with other observations in previous and present work cited above, constitutes a considerable body of evidence indicating that the stress-corrosion cracking of AZ61X magnesium alloy is strongly associated in some way with plastic deformation.

The hypothesis given in reference 6 that the plane of failure is determined by the directionality of the FeAl platelets cannot apply to the present data which suggest that mechanical rather than electrochemical factors determine this plane. However, it is conceivable that the presence of FeAl platelets lying parallel to the basal plane is responsible for the fact that a mechanism of basal failure exists.

Columbia University,
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TABLE I

EFFECT OF SOLUTION-ANNEALING TEMPERATURE ON SUSCEPTIBILITY TO STRESS
 CORROSION OF DUPLICATE STRIPS OF POLYCRYSTALLINE AZ61X MAGNESIUM
 ALLOY, INITIALLY STRESSED ABOVE YIELD STRENGTH, IN STANDARD
 (35 GRAMS SODIUM CHLORIDE PLUS 20 GRAMS POTASSIUM
 CHROMATE) SALT-CHROMATE SOLUTION

Treatment	Time for visible crack to form, min
440° C for 16 hr	6; 12
440° C for 16 hr + 370° C for 24 hr	6; 8
440° C for 16 hr + 300° C for 32 hr	9; 7,000
410° C for 24 hr	7; 13
390° C for 24 hr	17; 22
370° C for 24 hr	^a 9
350° C for 24 hr	6; 8
330° C for 24 hr	7; 28

^aSpecimen was unbroken after 3 days at which time stress was increased and it broke in 14 min.

TABLE II

STRESS-CORROSION BEHAVIOR IN VARIOUS CORRODENTS OF DUPLICATE STRIPS
 OF POLYCRYSTALLINE AZ61X MAGNESIUM ALLOY, INITIALLY
 STRESSED ABOVE YIELD STRENGTH, QUENCHED
 AFTER 24-HOUR ANNEAL AT 350° C

Corrodent	First crack observed, hr	Specimen fails, hr	Number of cracks formed
Standard salt-chromate solution	1/10; 2/15	13/60; 1/6	One or two
Distilled water	48; 84	60; 96	Several
0.5 percent hydrofluoric acid	24; 24	36; 72	Many
1.5 percent hydrofluoric acid	<1; 24	(a)	Many
5 percent hydrofluoric acid	1; 1	24; (a)	Many
.25 percent potassium fluoride	24; 120	96; (b)	Few
.5 percent hydrofluoric acid + 1 percent potassium fluoride	8; 8	168 to 216; (b)	Few

^aCracks only progressing very slowly after 48 to 72 hr.

^bCracks not progressing noticeably at time companion specimen broke.

TABLE III
STRESS-CORROSION TESTS OF SINGLE CRYSTALS OR COARSE-GRAINED
SPECIMENS OF AZ61X MAGNESIUM ALLOY

(a) Data for specimens having more than one grain in test length showing
that failure occurs in grain which suffers plastic deformation

Specimen	Condition (a)	Corrodent	Grain orientation, χ_0 , deg	Stress at failure, psi	χ_0 of failure grain, deg	Visual observations on specimen surface
6	AC	Distilled water	1; 8	^b 5,500	8	^c Failure grain not greatly distorted; irregular transverse failure
7	AC	Distilled water	6; 20; 61	4,300	61	^c Distortion of failure grain evident; initial crack runs at high angle to basal plane
23	AC	1 percent hydrofluoric acid	29; 71	3,650	29	^c Distortion of grain of failure evident; surface shows basal slip traces and some cross markings; fracture started at high angle to basal plane; 71° grain shows few markings which appear to be twins
12	WQ	0.5 percent hydrofluoric acid	5; 75	4,500	75	Failure grain shows little distortion and some strain markings which appear to be twin bands; failure started par- allel or nearly parallel to basal plane; 5° grain is not distorted but shows some strain markings

^aAC indicates air-cooled from 480° C; WQ indicates water-quenched from 350° C.

^bSpecimen was loaded as follows: 2,400 psi for 17 hr; 5,250 psi for 15 hr; 5,500 psi, failed in 10 hr.

^cIt was verified by X-ray photographs that there was considerable distortion of lattice in grain of failure and very little distortion in other grains (see fig. 4).

TABLE III.- Concluded
STRESS-CORROSION TESTS OF SINGLE CRYSTALS OR COARSE-GRAINED
SPECIMENS OF AZ61X MAGNESIUM ALLOY

(b) Data for specimens having comparable orientations showing that behavior does not vary greatly in different corrodents employed

Specimen (a)	Condition (b)	Grain orientation, χ_o , deg	Corrodent	Stress at failure, psi	Remarks
4	AC	31	Distilled water	^c 4,200	Number of small edge cracks
19	WQ	34	0.5 percent hydrofluoric acid	4,000	Number of small edge cracks
23	AC	^d 29	1 percent hydrofluoric acid	3,650	Number of small edge cracks
32	WQ	37	Salt-chromate solution	^e 3,500	Single-stepped crack with one branch; branch is parallel to or nearly parallel to basal plane
33	WQ	32	Petrolatum	>7,500	No cracks observed although specimen was much more severely deformed than others in table

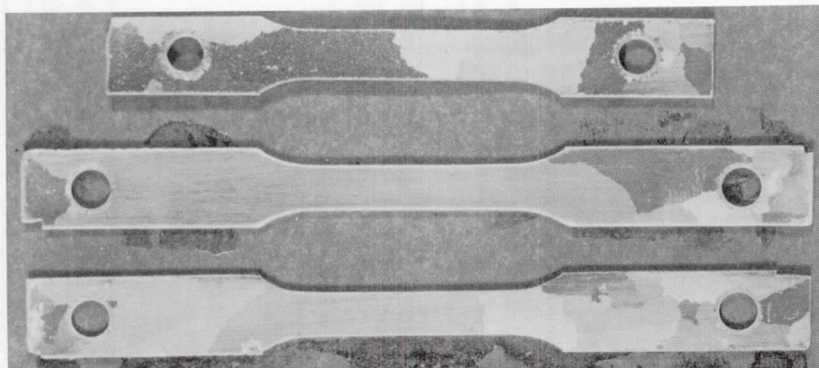
^aIn each of these specimens (except 33) average plane of initial part of failure runs at angle estimated to be 70° to 90° from basal plane; all show strong basal slipbands, sometimes bent, and strain markings running across basal slip traces.

^bAC indicates air-cooled from 480° C; WQ indicates water-quenched from 350° C.

^cSpecimen 4 was stressed directly to 4,200 psi and failed within 44 hr.

^dTwo-grain specimen which failed in 29° grain.

^eSpecimen 32 was stressed 2 days at 2,000 psi, 2 days at 3,000 psi, and failed after 1/2 hr at 3,500 psi.



L-57-2554

Figure 1.- Grain sizes of several types of test specimens. Top, three grains in reduced section (specimens actually used had grains of approximately equal length); middle, one grain; bottom, small grain would be masked out to expose only one grain to corrosive; picral-acetic-acid etch. Magnification, 1X.

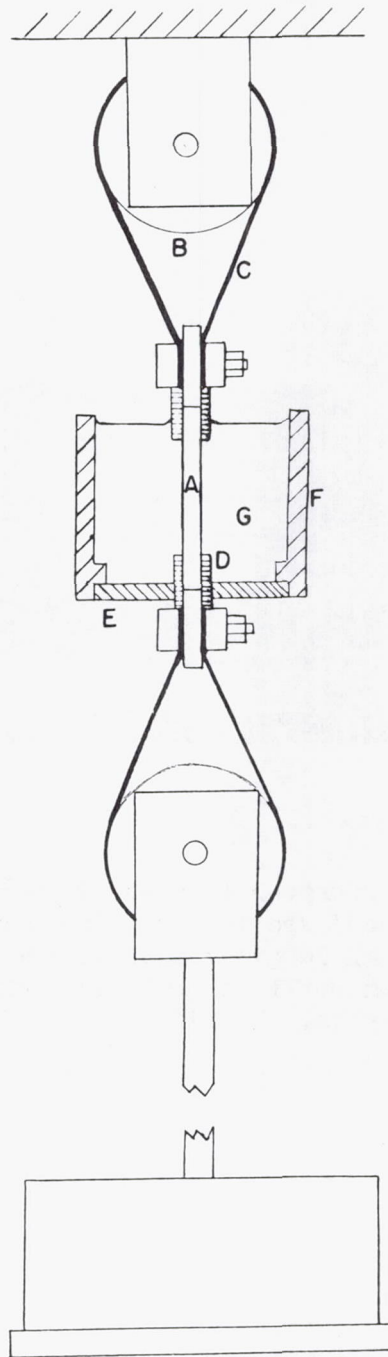
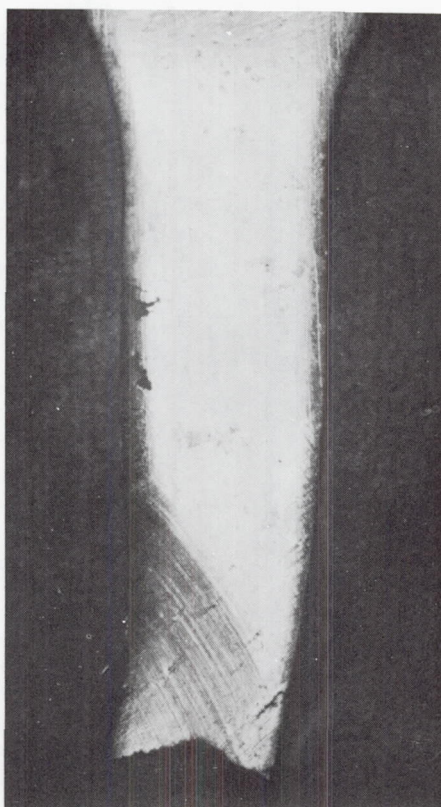
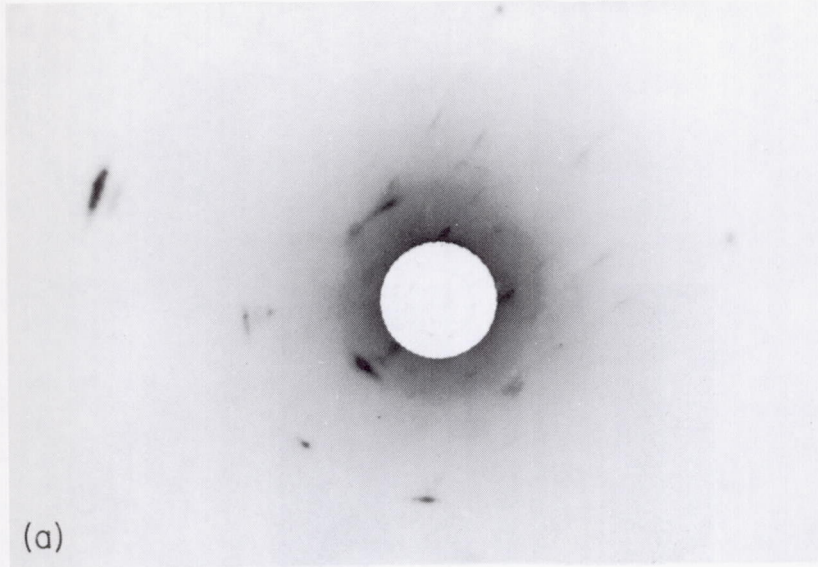


Figure 2.- Side view of stress-corrosion test apparatus. Container for corrodent is shown in section. Letters indicate: A, specimen; B, self-aligning ball bearings; C, steel strips; D, beeswax-rosin masking; E, neoprene plug; F, acrylic cup; and G, corrodent.

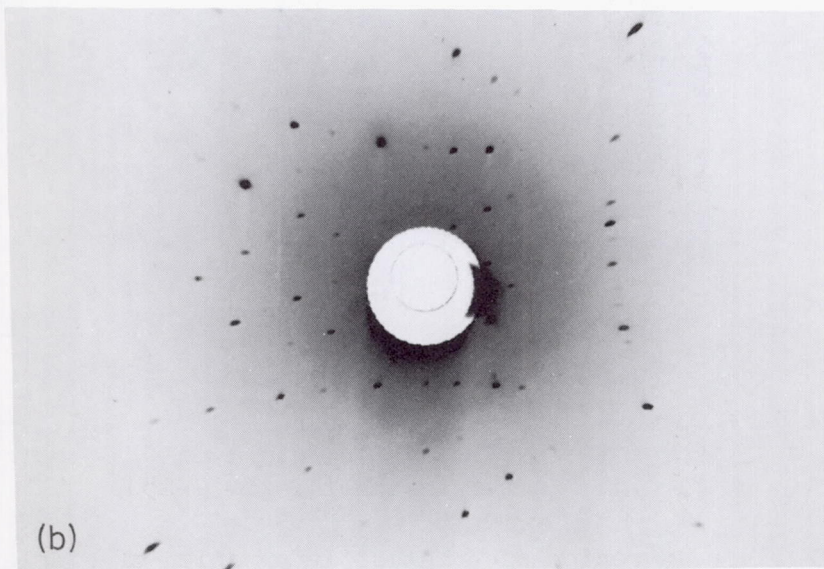


L-57-2555

Figure 3.- Failure in region of greatest deformation of single crystal (specimen 4) which had deformed considerably at 4,200 psi before cracking (starting at left edge) in distilled water. Bent basal slipbands (grain orientation, χ_0 , 31°) run from upper left to lower right. Magnification, 4.5X.



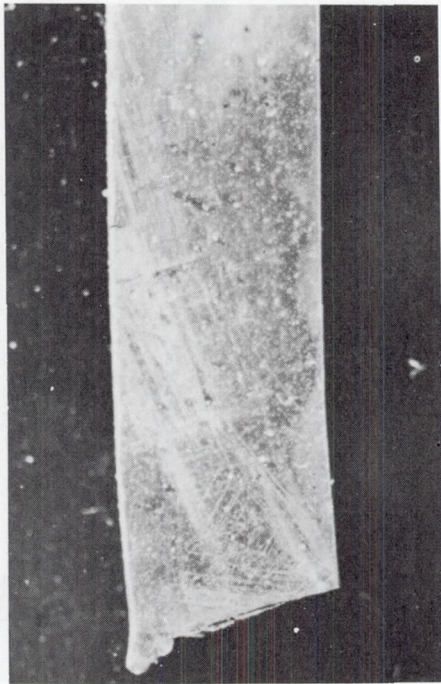
(a) Failure grain 3 millimeters from failure surface; severe lattice distortion and probably twinning.



L-57-2556

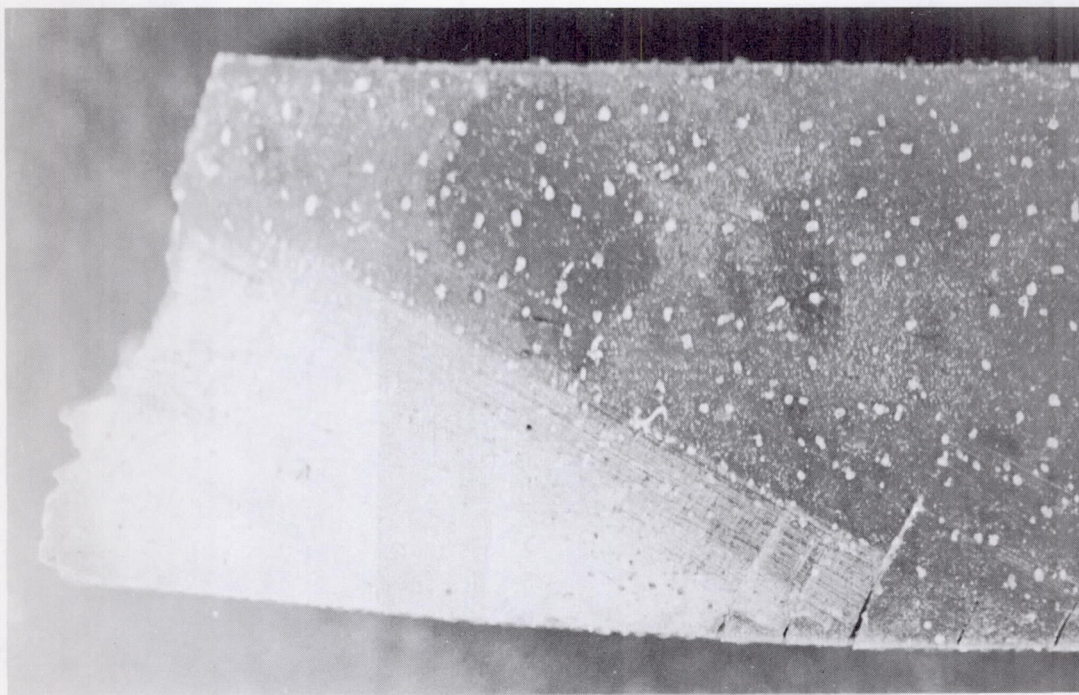
(b) Grain which remained intact; very little or no lattice distortion produced by stress applied during test.

Figure 4.- Back-reflection Laue X-ray photographs of two grains of specimen 7 after stress-corrosion failure. Tungsten radiation; diameter irradiated about 1 millimeter; specimen-to-film distance, 3 centimeters.



L-57-2557

Figure 5.- Failure which started at right parallel to basal plane in lower (75°) grain of two-grain specimen which failed at 4,500 psi in 0.5 percent hydrofluoric acid. Magnification, 4.5X.



L-57-2558

Figure 6.- Region near failure of specimen 23, which failed in 29° grain in 1 percent hydrofluoric acid at 3,650 psi, showing that edge cracks crossing basal slipbands (coarse bands are visible and run from upper left to lower right) are often associated with deformation markings which run across the basal slipbands. Failure began at upper edge. Magnification, 12X.